NASA CR-54314

PREPARATION and EVALUATION of FIBER METAL NICKEL BATTERY PLAQUES

SECOND QUARTERLY PROGRESS REPORT

November 1, 1964 to January 31, 1965

by

J. L. Bidler and J. I. Fisher

prepared for

NATIONAL AERONAUTICS and SPACE ADMINISTRATION

CONTRACT NAS 3-6006

HUYCK METALS

A DEPARTMENT OF HUYCK CORPORATION

NASA CR-54314

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I. SUMMARY

Internal surface area, tensile strength, and electrical resistivity measurements have been made for AX1 and AX2 nickel fiber metal battery plaques sintered at 1600°F, 1800°F, 2000°F and 2150°F for 20 minutes in hydrogen. The results obtained define the sintering parameters to be used to produce nickel fiber metal battery plaques with maximum tensile strength and internal surface area and minimum electrical resistivity.

The internal surface area decreases with increasing sintering temperature from a maximum of 440 cm^2/gm for AX1 nickel fiber sintered at 1600°F to a minimum of 330 cm^2/gm for AX2 nickel fiber sintered at 2150°F.

The tensile strength of 10% dense AX1 nickel fiber plaques is greater than that of 10% dense AX2 nickel fiber plaques at all sintering temperatures studied except 1600°F where the tensile strengths were similar.

The tensile strengths and the difference in tensile strengths increase as the sintering temperature increases. The maximum tensile strengths measured were 295 psi for AX1 nickel fiber plaques and 209 psi for AX2 nickel fiber plaques both sintered at 2150°F.

The electrical resistivity of 15% dense AX2 nickel fiber plaques decrease as the sintering temperature increases from 814 microhm-cm at 1600°F to 315 microhm-cm at 2150°F. The electrical resistivity of 15% dense AX1 nickel fiber plaques decreases from 1090 microhm-cm at 1600°F to 416 microhm-cm at 1800°F then increases to 653 microhm-cm at 2000°F before decreasing again to 324 microhm-cm at 2150°F. This unexpected increase in electrical resistivity cannot be explained at present. The experiment is being repeated in an attempt to verify the results.

The data accumulated to date indicate that optimum plaque characteristics will be obtained by sintering between 1800°F and 2000°F for 20 minutes. Future work will include sintering AX1 nickel fiber plaques at 1850°F, 1900°F and 1950°F to determine the maximum temperature to maximize the internal surface area and minimize the electrical resistivity.

II. INTRODUCTION

This program, for the preparation and evaluation of nickel fiber metal battery plaques, is intended to provide a material showing substantial improvement over existing plaques. The virtues of using metal fibers for the production of battery plaques are:

1. Porosity Range

   A wider porosity range can be achieved using fibers than with any other particle form.

2. Control of Pore Size

   Fiber diameter and plaque density interact to define pore size. Using the fibers selected for this program, pore sizes ranging from 10 to 80 microns can be obtained at high porosity levels.

3. Control of Pore Size Distribution

   The normal procedure for manufacturing fiber metal is to felt and sinter to a high porosity, then compact to the desired porosity. This affords precise control of the pore size range which decreases as the porosity decreases.

4. Maximum Interconnected Porosity

   Fiber metal structures with porosities as low as 50% have more than 95% interconnected pores.

5. Large Surface Area

   Fiber size and shape interact to define surface area. The fibers employed in this study have specific surface areas in excess of 450 cm^2/gm.

6. Strength

   Fiber metal bodies have the highest strength of any porous material at high porosities.

7. Formability

The compressibility of metal fiber materials permits considerable latitude in forming operations.

OBJECTIVES

The objective of this program is the development of nickel fiber metal battery plaques having minimum density and electrical resistivity and maximum internal surface area, strength, and flexibility. The primary materials to be evaluated are two grades of nickel fiber differing in apparent diameter. The processing parameters necessary to optimize the aforementioned plaque properties are to be defined and sample plaques produced and characterized.

PROGRAM OUTLINE

The program outline defines four major Tasks, A-D, which are summarized as follows:

Task A. Raw Material Classification

Each raw material used in the program is to be characterized as to particle shape, particle size, and particle size distribution. Microscopic measurements of fiber lengths and diameter are to be supplemented by photomicrographs of as-sintered surfaces, cross sectional areas, and shadographs of typical fibers to present both a statistical and a visual description.

Task B. Sintering Study

It is desired to establish the highest sintering temperature that will produce an acceptable amount of shrinkage when the sintering time is held constant at 20 minutes. The sintered plaques resulting from this phase of the program will be evaluated to determine the median pore size, pore size distribution, density, tensile strength, internal surface area, and electrical resistivity.

These data will be used to define the sintering temperature to be used to produce plaques with the desired characteristics.

Task C. Plaque Classification

Plaques processed under the conditions defined in Task B. will be produced and electrical resistivity, internal surface area, density, median pore size, pore size distribution, tensile strength and flexibility will be determined.

Task D. Plaque Samples

A sample of each test plaque upon which the classification tests were performed will be provided to the NASA Project Manager.

III. EXPERIMENTAL PROCEDURES AND APPARATUS

Task A. Raw Material Classification

The techniques and apparatus used and the results obtained for the raw materials classified were presented in the First Quarterly Progress Report.

Task B. Sintering Study

The procedures and apparatus used to determine the effect of sintering temperature upon the density, median pore size and pore size distribution were presented in the First Quarterly Progress Report.

1. Internal Surface Area Measurements

Apparatus

An air permeability apparatus is employed for surface area determinations. This equipment is designed to measure accurately the pressure drop across a permeable sample when the sample is exposed to a calibrated flow of air.

Procedure

The method used to determine the internal surface area of nickel fiber metal plaques is that described by Orr and Dallavalle[1] wherein the pressure drop of a calibrated flow of air through a bed of fine, fibrous, packed material can be related to the specific surface area of the material by means of the Kozeny-Carman equation:

$$S_v^2 = \frac{g_c}{K\mu V} \quad \frac{(\Delta P)}{L} \quad \frac{\epsilon^3}{(1-\epsilon)^2}$$

where S_v = Specific surface area of solids, surface area/unit volume of solids present

g_c = gravitational constant

μ = viscosity of flowing fluid

V = velocity of flowing fluid

ΔP = pressure drop through packed bed

L = length of packed bed

ϵ = porosity; void volume/total packed bed volume

K = Kozeny constant = 4.5 for spheres, 3.0 for cylinders arranged parallel to flow, 6.0 for cylinders arranged perpendicular to flow.

This method is reliable if the permeability data are taken in the streamline region where the flow rate varies linearly with the pressure drop.

(1) Superscripts refer to similarly numbered entries in the bibliography.

Using air under the conditions of streamline flow and samples of constant porosity the Kozeny-Carman equation can be reduced to

$$S_V^2 = K_2 \frac{\Delta P}{VL}$$

The constant K_2 can be evaluated by obtaining permeability coefficients ($\frac{\Delta P}{VL}$) for samples of known surface area.

For this investigation, an average value of K_2 was calculated by obtaining permeability coefficients for sample plaques made from wire of 0.003, 0.004 and 0.006 inch diameter.

The samples of AX1 and AX2 nickel fiber plaques sintered at various temperatures were tested at densities of 20% and 30% of theoretical density. At higher porosities the flow rate required to produce accurately measurable pressure drops across thin samples is in excess of 3000 SCFH/ ft^2 (standard cubic feet per hour per ft^2), which has been shown to be the upper limit for streamline flow. Since the method used to increase the density is a simple mechanical compaction, the specific surface area is not significantly changed.

Samples of AX1 and AX2 nickel fiber plaques were placed in a sample holder designed to eliminate edge effects. A stream of air at 30 psi was passed through each sample at a rate that would produce a pressure drop of 0.1 or 1.0 inches of H_2O. The flow rate was controlled by one of six calibrated orifices, depending upon the flow rate required, and measured by means of a mercury manometer, which determines the pressure drop across the selected orifice.

The pressure drop across the sample was read from an inclined water gage manometer, calibrated in hundredths of inches.

Permeability coefficients were obtained at 20% and 30% of theoretical density for AX1 and AX2 nickel fiber metal plaques sintered at 1600°F, 1800°F, 2000°F and 2150°F for 20 minutes.

2. Tensile Strength Measurements

Apparatus

Hounsfield Tensometer with 62.5 pound beam. TensilkuT milling machine with ASTM standard E8-54T tensile specimen fixture.

Procedure

Procedure is in accordance with ASTM standard E8-54T.

Plaques of AX1 and AX2 nickel fiber sintered at 1600°F, 1800°F, 2000°F and 2150°F for 20 minutes were impregnated with a low melting point (275°F) salt to prevent damage to the macrostructure during machining. The salt impregnated plaques were sawed into 2 inch by 6-1/2 inch strips and tensile specimens were milled using a TensilkuT mill and fixture. The tensile specimens had a 2 inch gauge length, were 1/2 inch wide and approximately 0.060 inches thick.

The salt was leached from the specimens using warm water; the specimens were dried and pulled with a Hounsfield Tensometer. The load required to break each specimen was recorded and the original cross sectional area was used to calculate the tensile strength. Duplicate specimens were tested for each material at each sintering temperature.

3. Electrical Resistivity Measurements

Apparatus

Leeds and Northrup Kelvin Bridge Model number 4288.

Procedure

Samples of plaques of AX1 and AX2 nickel fiber 1/2 inch wide by 12 inches long by approximately 0.060 thick were prepared as described above for the preparation of tensile specimens. The cut edges were surface ground to eliminate spurious edge effects due to compaction or smearing, and to obtain precise width.

The nickel fiber metal strips were clamped securely in a sample holder designed to compact the fiber metal between two $\frac{1}{2}$" radii at each end of the strip. The compacting assured contact and constant contact resistance. The length, which was measured between the line contacts of the radii, resistance, and cross sectional area of each strip was measured and recorded. Duplicate samples, when available, of each material at each sintering temperature were tested. Procedures and data reporting are in accordance with ASTM standard A344-64

IV. EXPERIMENTAL RESULTS AND DISCUSSION

Task A. Raw Material Classification

Table I summarizes the data that were obtained and presented in the First Quarterly Progress Report.

TABLE I

Summary of Particles Size and Particle Size Distribution Data

Material	Diameter - Microns				Length - Microns			
	Mean	Median	Standard Deviation	Range	Mean	Median	Standard Deviation	Range
AX1	11.2	5-7.5	10.3	1-50	139	88-112	163	13-1500
AX2	14.9	10-12.5	11.5	1-50	159	88-112	178	13-1500

Task B. Sintering Study

1. Internal Surface Area Measurements

The permeability coefficients of plaques made from 0.003, 0.004 and 0.006 inch diameter wire are shown in Table II with the average value of the constant K_2.

TABLE II

Permeability Coefficients, Surface Area and Orientation Factor for Plaques Made from Wire.

	Permeability coefficient $\frac{VL}{P}$ SCFH/FT^2/in H_2O/in thickness	Known surface area cm^2/gm3	K_2
30% dense 0.003" wire	2150	525	6.0×10^8
30% dense 0.004" wire	2000	394	7.8×10^8
30% dense 0.006" wire	8300	262	5.6×10^8
Average			6.5×10^8

The permeability coefficients (VL and ΔP) obtained for AX1 and AX2 nickel fiber plaque at two densities are shown in Table III. Fig. 1 and Fig. 2 are semi-logarithmic plots of density versus flow rate at a constant pressure drop. To keep the various parameters in compatible units and to calculate the surface area at the same density that the K_2 constant was derived at, it is necessary to determine the flow rate at a density of 30% of theoretical and the thickness at one inch. The plots of flow versus density are essentially parallel at pressure drops of 0.1 and 1.0 inches of H_2O; therefore the pressure drop of 0.1 inches of H_2O was used to calculate the internal surface area. The data presented in Table IV were

TABLE III

Flow Rate at Pressure Drops of 0.1 and 1.0 Inch of H_2O for AX1 and AX2 Nickel Fiber Plaques

Material	Sintering Temp. °F	Density % of Theoretical	Flow Rate SCFH/ft^2 at ΔP = 0.1 inch H_2O	Flow Rate SCFH/ft^2 at ΔP = 1.0 inch H_2O
AX1	1600	18.1	420	4180
	1600	27.8	178	1720
	1800	19.3	417	4280
	1800	28.2	172	1880
	2000	19.2	500	4780
	2000	31.6	209	2240
	2150	19.4	570	5250
	2150	28.6	308	3220
AX2	1600	18.1	625	5800
	1600	28.3	224	2380
	1800	18.6	520	5915
	1800	28.8	234	2500
	2000	19.0	625	6100
	2000	29.8	266	2780
	2150	19.3	705	6500
	2150	28.8	334	3375

FIGURE 1

Flow Rate vs Density and Sintering Temperature for AX1 Nickel at a Pressure Drop of 0.1 inch of H_2O and a Thickness of 0.030 inch.

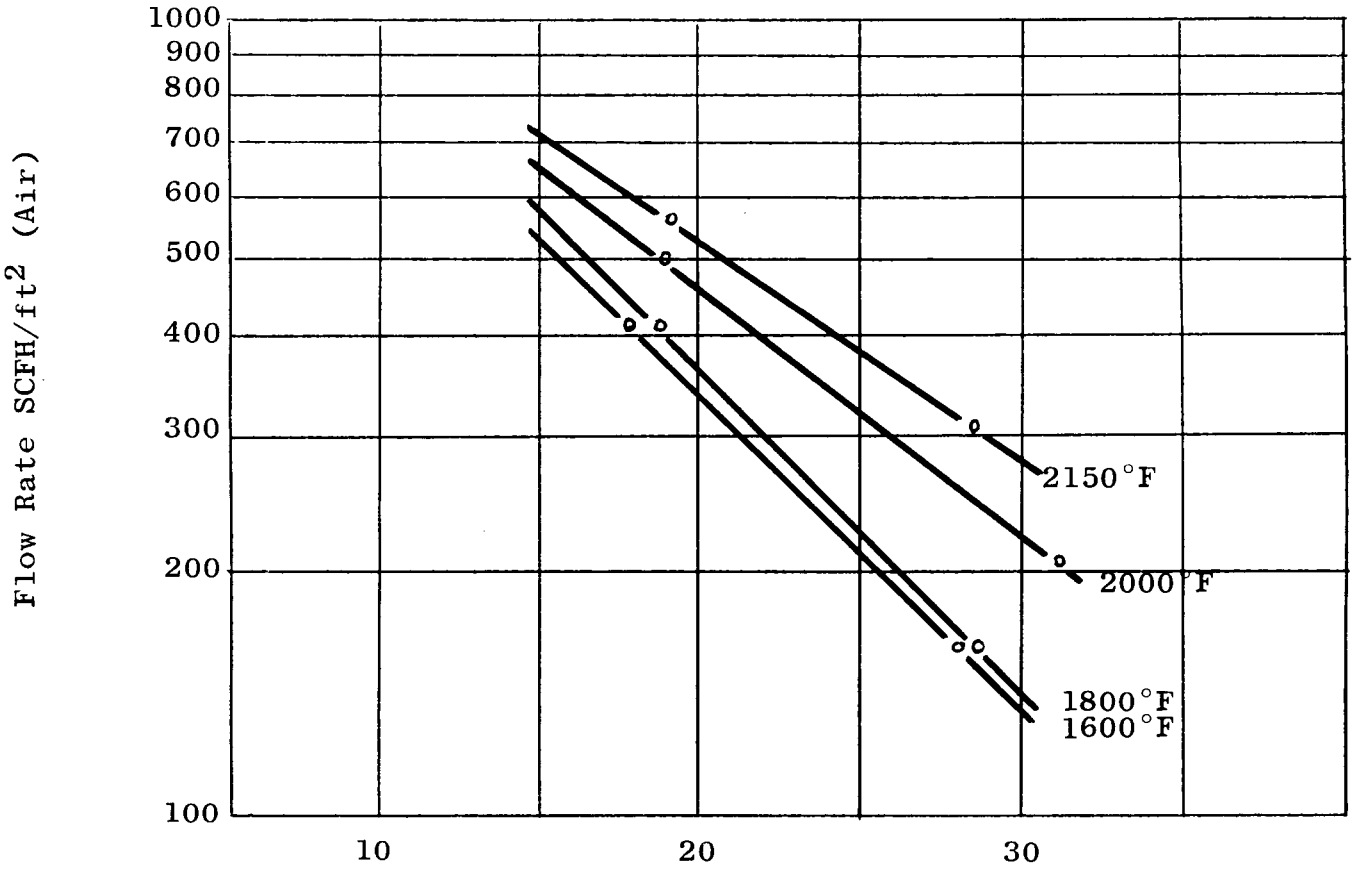
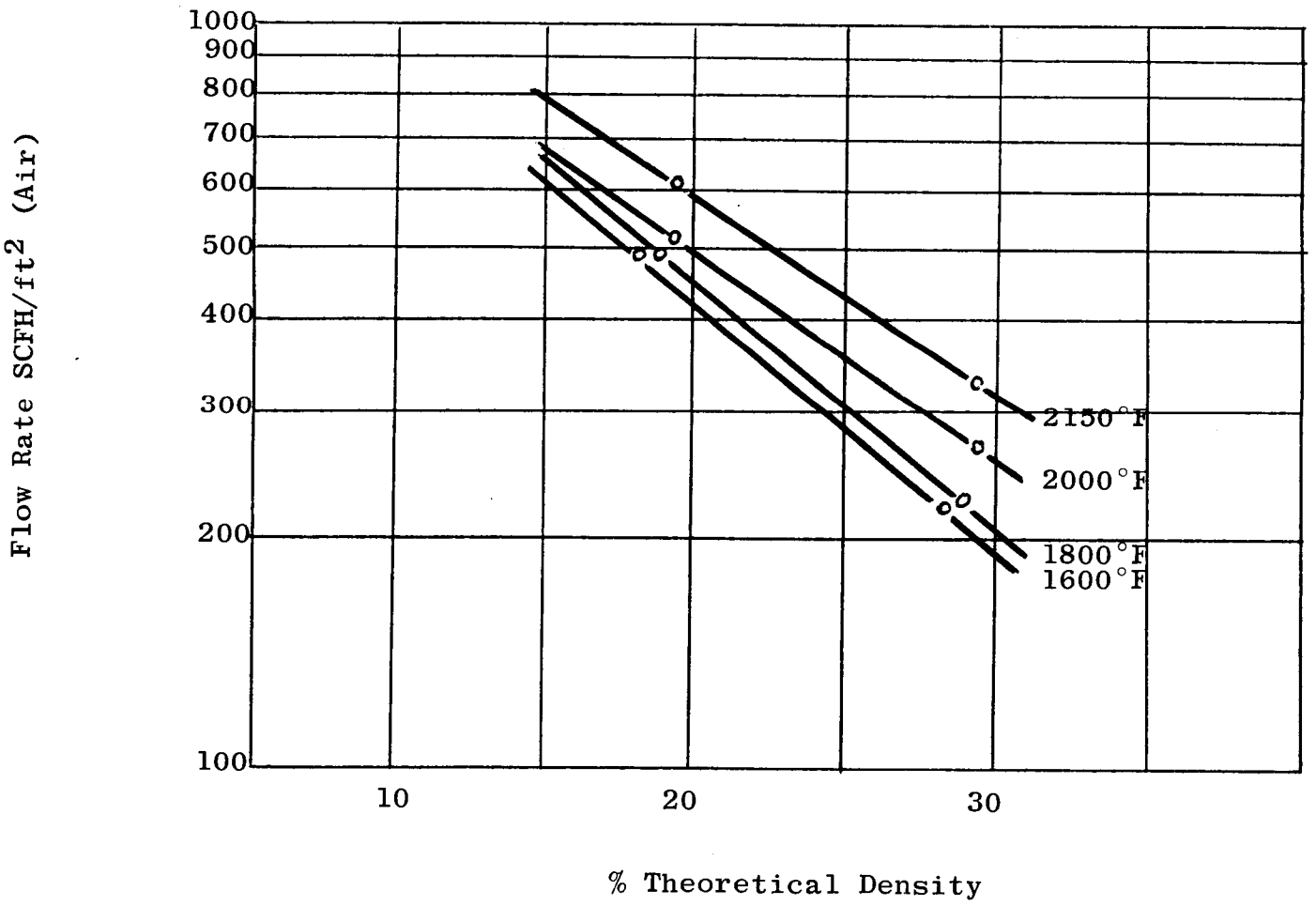


FIGURE 2

Flow Rate vs Density and Sintering Temperature for AX2 Nickel at a Pressure Drop of 0.1 inch of H_2O and a Thickness of 0.030 inch.



calculated using a density of 30% and a pressure drop of 0.1 inches of H_2O.

The specific surface area in cm^2/gm is obtained by dividing the specific surface area in cm^2/cm^3 by the density of nickel which is 8.9 gm/cm^3.

TABLE IV

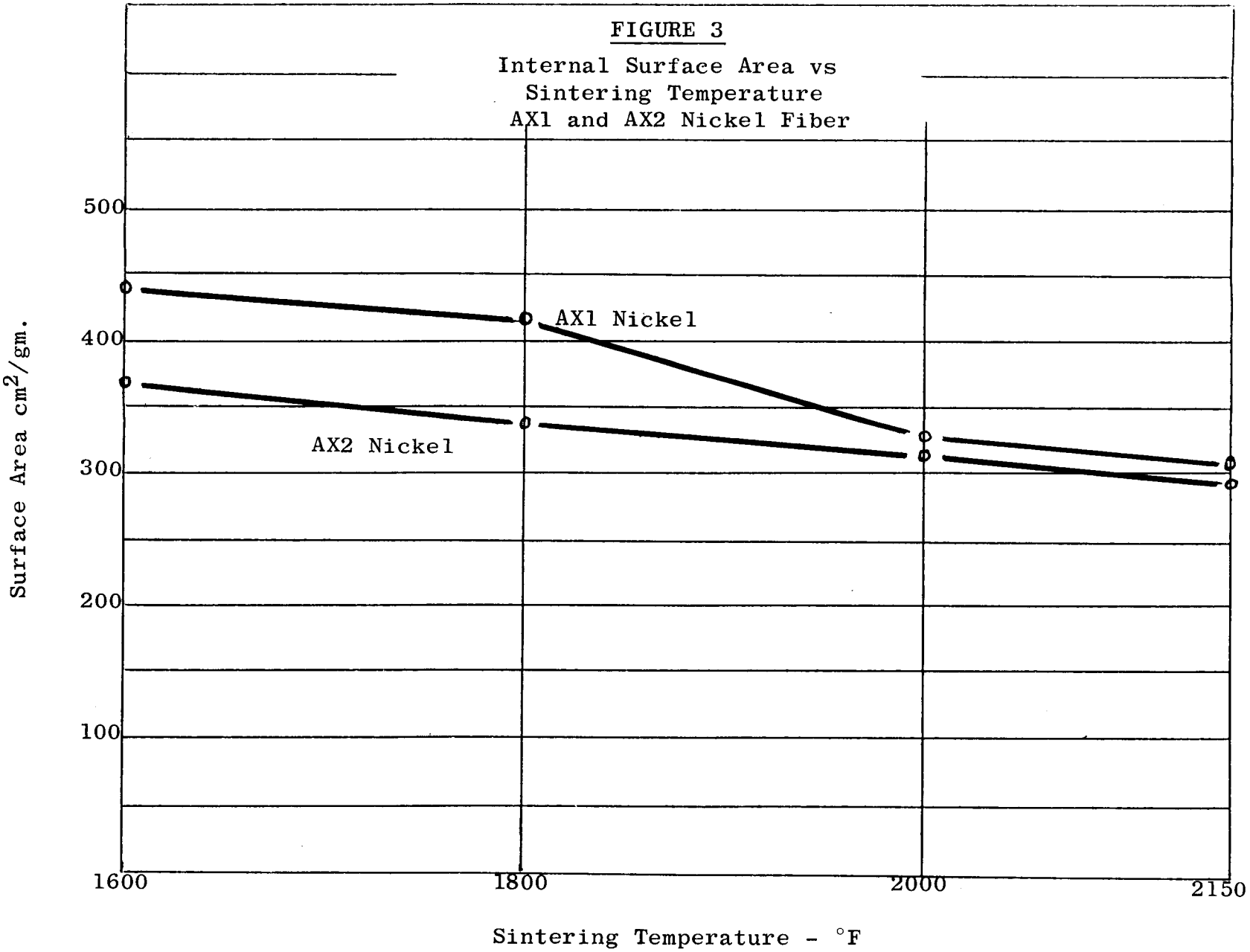
Permeability Coefficients and Specific Surface Area of AX1 and AX2 Nickel Fiber Metal Plaques Sintered at Various Temperatures

Material	Sintering Temp. °F	$\frac{VL}{\Delta P}$ SCFH/ft^2/ in H_2O/in Thickness	S_v^2	$S_v \frac{cm^2}{cm^3}$	$S_v \frac{cm^2}{gm}$
AX1	1600	42	15.4×10^6	3920	440
	1800	46.5	13.9×10^6	3730	419
	2000	75	8.6×10^6	2930	329
	2150	90	7.2×10^6	2690	302
AX2	1600	60	10.8×10^6	3280	369
	1800	69.5	9.3×10^6	3050	343
	2000	82.5	7.8×10^6	2800	315
	2150	93	6.95×10^6	2640	296

A graph of internal surface area versus sintering temperature, Figure 3, shows that AX1 has a greater surface area than AX2 nickel fiber until the sintering temperature reaches 2150°F. The data presented in the First Quarterly Progress Report show that the apparent diameter of AX1 nickel fiber is approximately 30% smaller than that of AX2 nickel fiber. It is apparent from the surface area measurements that the "thickness", or the dimension of the fiber that lies in the viewing plane when observed through a microscope, must also be smaller for AX1 nickel fiber.

FIGURE 3

Internal Surface Area vs
Sintering Temperature
AX1 and AX2 Nickel Fiber



The greater surface area of AX1 nickel fiber renders this material more susceptible to sintering than a coarser fiber because the rate of sintering is sensitive to surface free energy. Consequently, at the same sintering temperature the AX1 fiber sinters more rapidly and the internal surface area is reduced to a greater degree. (2). Figures 4-7 and 8-11 are photomicrographs of AX1 and AX2 sintered at the indicated temperatures. As the sintering temperature increases, the reduction in surface energy is accelerated; the sharp edges of the fibers become more rounded and their cross section takes on a more symetrical shape. The internal surface area is therefore decreased.

2. Tensile Strength Measurements

The average tensile strength of AX1 and AX2 nickel fiber metal plaques sintered at the indicated temperatures is shown in Table V.

The density of the samples shown in Table V. varies as a result of the varying sintering temperature. It is known that the tensile strength increases in direct proportion to the density in the range from 10% to 20% density; consequently the strengths have been normalized linearly to 10% density. The deviation from linearity of the strength-density relationship is considered to be insignificant in the range of the tests.

The tensile strength of both materials is plotted as a function of sintering temperature in Figure 12. The tensile strength of AX1 nickel fiber is greater than that of AX2 nickel fiber at all sintering temperatures studied except 1600°F where the strengths are similar.

Fig. 4 Photomicrograph of AX1 Nickel fiber plaque sintered at 1600°F for 20 min. X210

Fig. 5 Photomicrograph of AX1 Nickel fiber plaque sintered at 1800°F for 20 min. X210

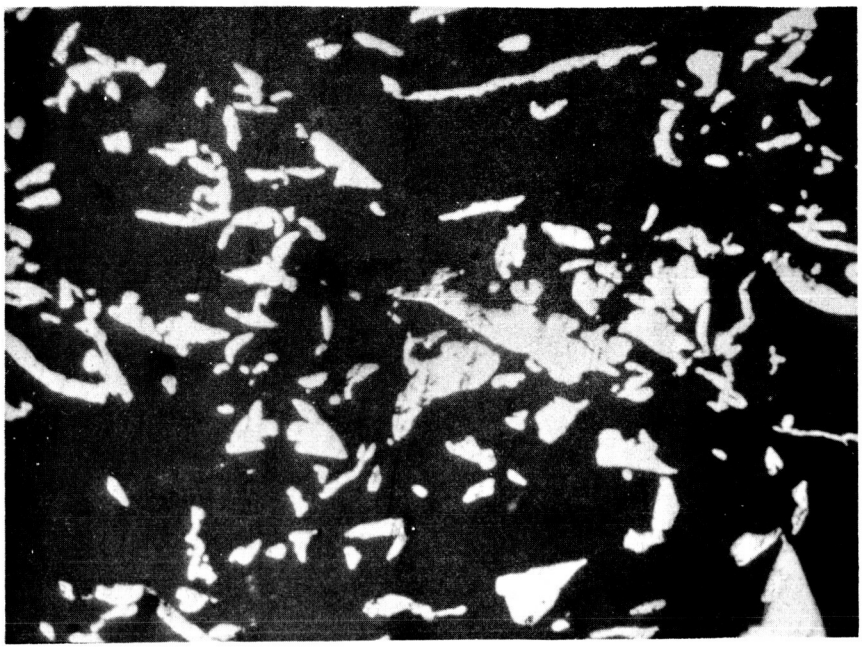


Fig. 6 Photomicrograph of AX1 Nickel fiber plaque sintered at 2000°F for 20 min. X210

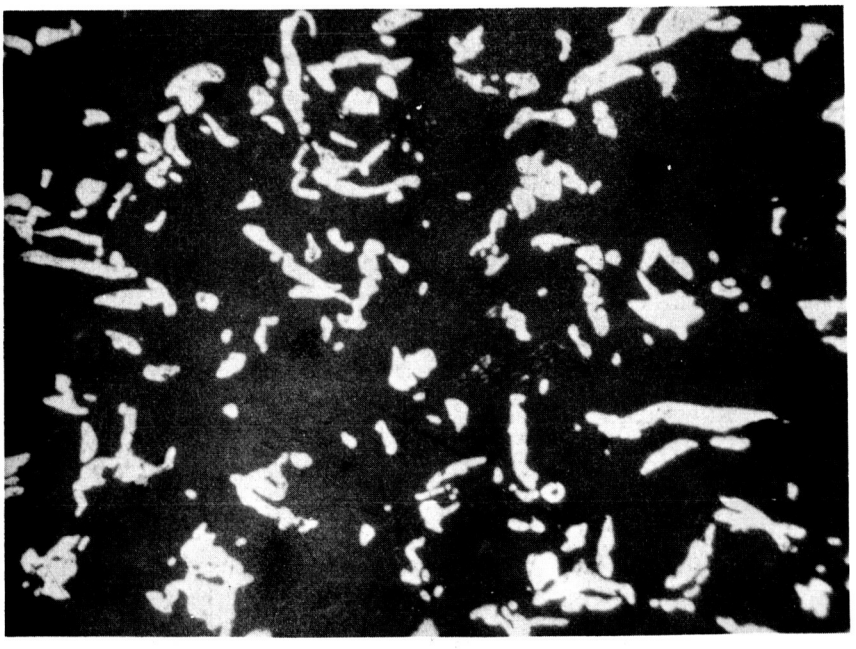


Fig. 7 Photomicrograph of AX1 Nickel fiber plaque sintered at 2150°F for 20 min. X210

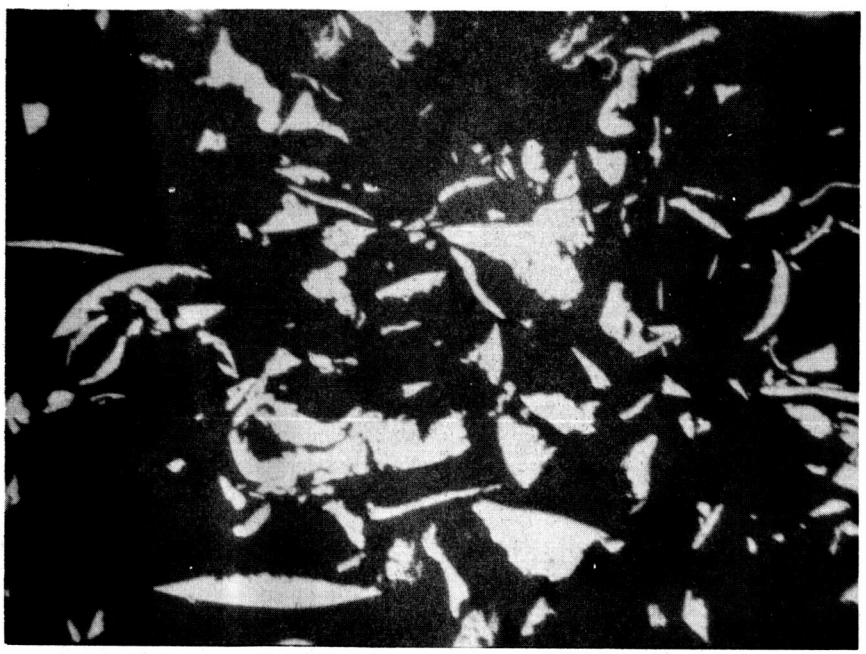


Fig. 8 Photomicrograph of AX2 Nickel fiber plaque sintered at 1600°F for 20 min. X210

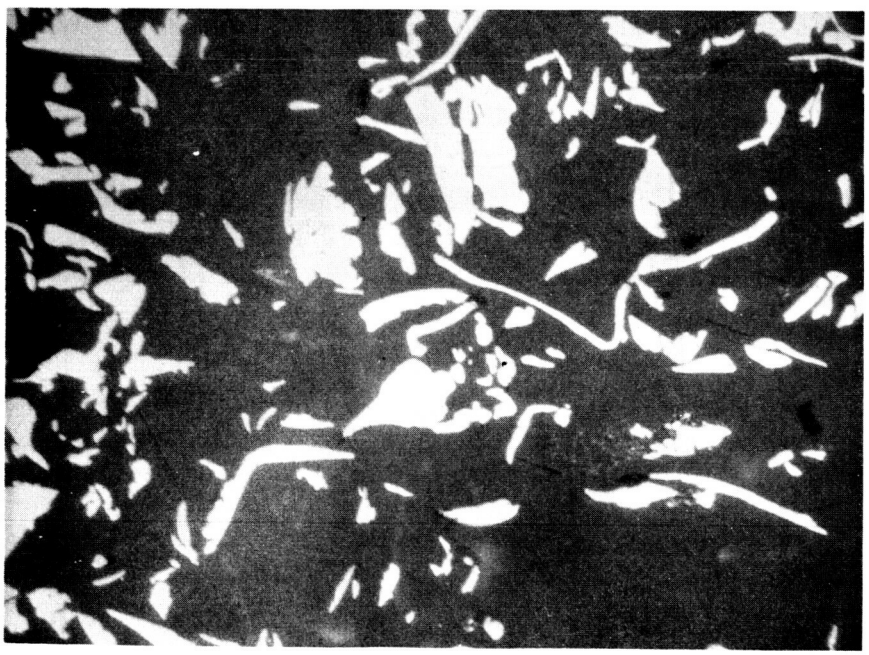


Fig. 9 Photomicrograph of AX2 Nickel fiber plaque sintered at 1800°F for 20 min. X210

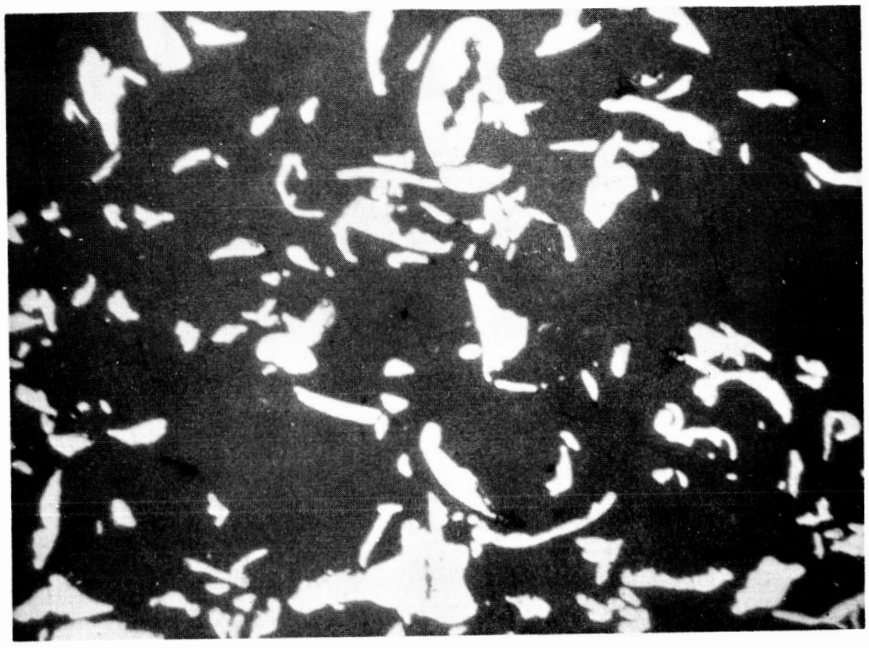


Fig. 10 Photomicrograph of AX2 Nickel fiber plaque sintered at 2000°F for 20 min. X210

Fig. 11 Photomicrograph of AX2 Nickel fiber plaque sintered at 2150°F for 20 min. X210

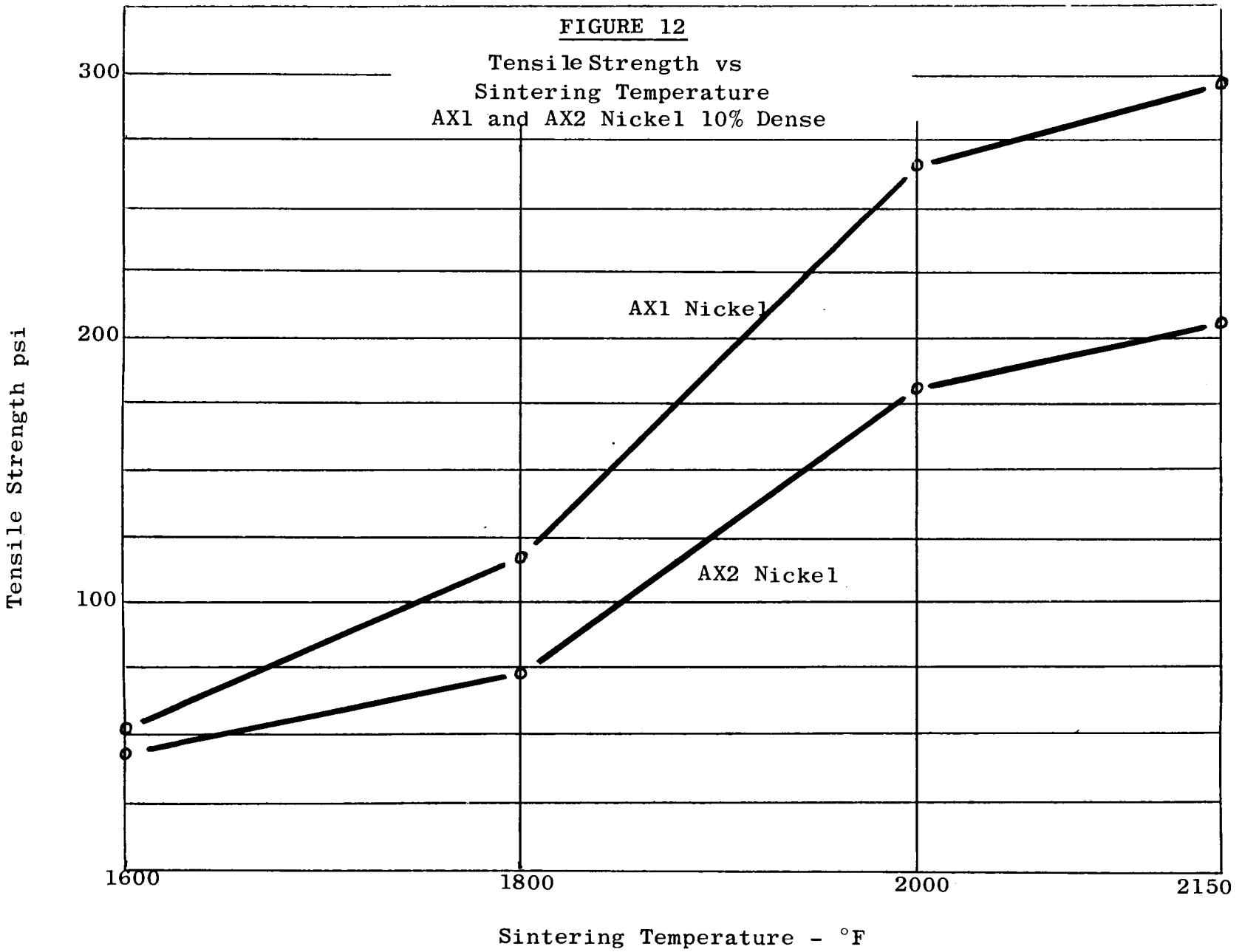
TABLE V.

Tensile Strength of 10% Dense AX1 and AX2 Nickel Fiber Plaques Sintered at Various Temperatures

Material	Sintering Temp. (degrees Fahrenheit)	Ultimate Tensile Strength (lbs. per sq. inch)	Average Ultimate Tensile Strength (lbs. per sq. inch)
AX1 Nickel 10% Dense	1600	41.2	41.2
	1800	114.0	119.0
	1800	124.0	119.0
	2000	264.0	265.0
	2000	273.0	265.0
	2000	257.0	265.0
	2150	292.0	295.0
	2150	298.0	295.0
AX2 Nickel 10% Dense	1600	56.5	50.5
	1600	44.5	50.5
	1800	74.0	74.4
	1800	77.0	74.4
	1800	72.2	74.4
	2000	168.0	181.0
	2000	187.0	181.0
	2000	188.0	181.0
	2150	209.0	209.0

FIGURE 12

Tensile Strength vs
Sintering Temperature
AX1 and AX2 Nickel 10% Dense



The greater surface area of AX1 than AX2 nickel fiber and the larger number of contact points at a given density reduce the time required to form sinter bonds of a size comparable to the cross section of the fiber. (3). Consequently, when AX1 and AX2 nickel fiber are sintered at the same temperature for the same length of time, the tensile strength of AX1 nickel fiber is greater. It would be expected that as time increased at any temperature, the strengths of the two materials would tend to converge.

3. Electrical Resistivity Measurements

The electrical resistivity of samples of AX1 and AX2 nickel fiber metal plaques sintered at the indicated temperatures is shown in Table VI. The reproducibility of the apparatus is indicated by the values obtained for duplicate or triplicate samples. The effect of contact resistance has been shown to be insignificant when leads of the proper resistance are used. This was verified by measuring the resistance of copper wire of different diameters and lengths.

TABLE VI.

Electrical Resistivity at 15% Dense AX1 and AX2
Nickel Fiber Plaques Sintered at Various Temperatures

Material	Sintering Temp. (degrees Fahrenheit)	Resistivity (microhm-cm)	Average Resistivity (microhm-cm)
AX1 Nickel 15% Dense	1600	1090	1090
	1800	416	416
	1800	416	416
	2000	656	653
	2000	650	653
	2150	328	324
	2150	321	324
	2150	322	324
AX2 Nickel 15% Dense	1600	784	778
	1600	773	778
	1800	445	433
	1800	429	433
	1800	425	433
	2000	357	354
	2000	355	354
	2000	351	354
	2150	318	315
	2150	311	315
	2150	315	315

As in the case of tensile strength samples, the density of each material varies with sintering temperature. The variation of electrical resistivity with density in the range from 10% to 20% dense has not been accurately measured for one sintering temperature. Nevertheless, the results were normalized to 15% dense material for comparison purposes. This density corresponds roughly to the median density of all the samples tested. It is possible that appreciable error could result from this normalization; additional verification work is planned.

A graph of electrical resistivity of 15% dense AX1 and AX2 nickel fiber plaques as a function of sintering temperature is presented in Figure 13. The electrical resistivity of AX1 nickel fiber metal plaques decreases from 1600°F to 1800°F then increases from 1800°F to 2000°F and decreases again between 2000°F and 2150°F. No explanation can be offered for this anomaly at present. In general, as the size of the sinter bond increases the resistivity would be expected to decrease until the fiber cross section becomes the limiting resistive element.

In an effort to resolve this anomaly, samples of AX1 plaques that were originally sintered at 1600°F, 1800°F and 2000°F respectively were resintered at 2000°F for 20 minutes. The resistivity of the resintered 1600°F sample decreased 33%, that of the 1800°F sample decreased 34%, and that of the 2000°F sample decreased 13%. These data tend to confirm the original findings. The initial experiment is currently being repeated in a further attempt to verify the result.

Task C. Plaque Classification

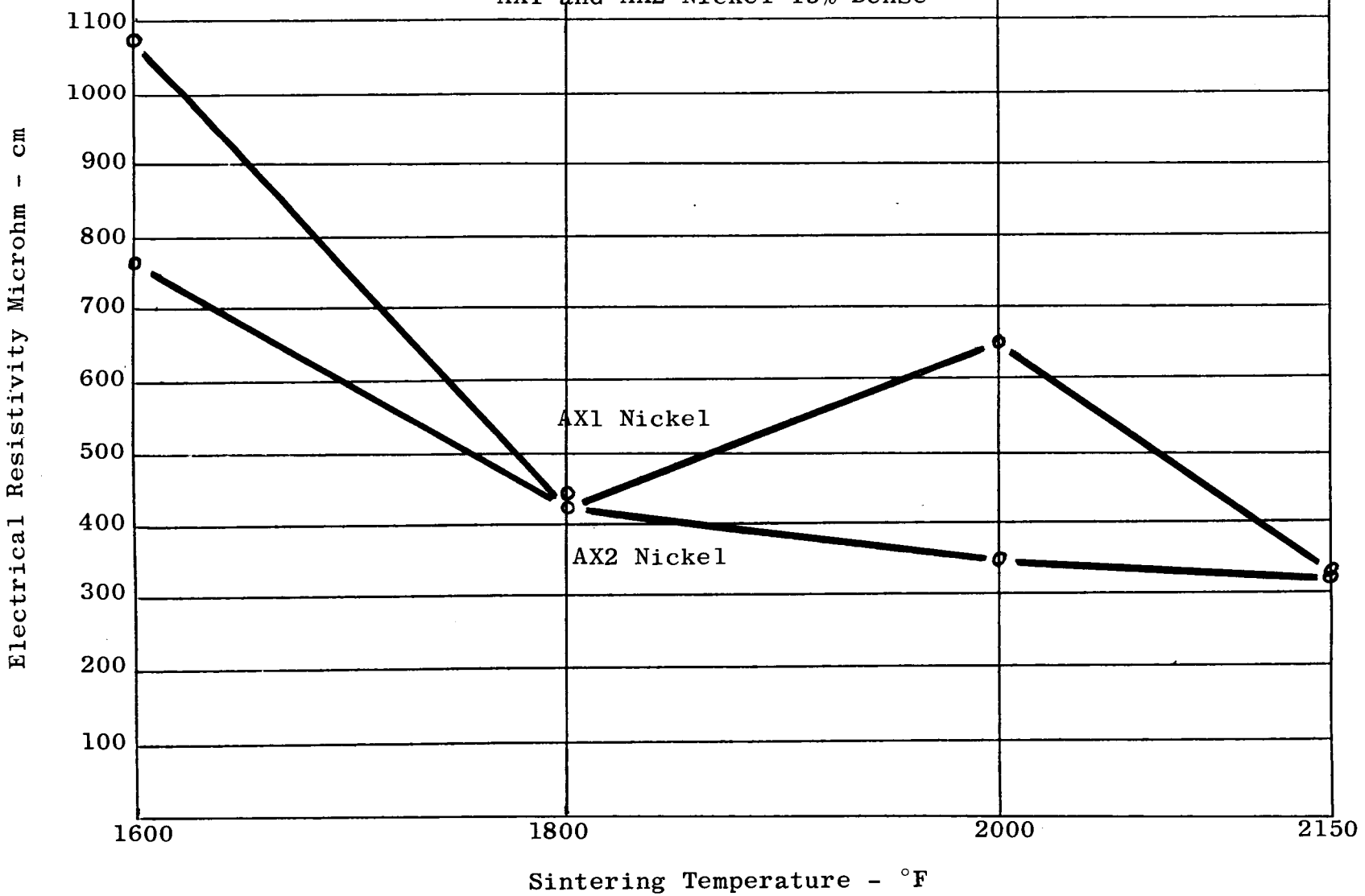
Classification tests on final configuration plaques have not been made to date.

Task D. Plaque Samples

Samples of plaques classified in Task C will be furnished when available.

FIGURE 13

Electrical Resistivity vs
Sintering Temperature
AX1 and AX2 Nickel 15% Dense



V. FUTURE WORK

Work during the next reporting period will be directed toward:

1. Redetermination of electrical resistivity versus sintering temperature for AX1 nickel fiber metal plaques.

2. Further investigation of the rate of decrease of surface area for AX1 nickel fiber metal plaque sintered in the temperature range from 1800°F to 2000°F.

3. Production of AX1 and AX2 nickel fiber metal plaques processed in accordance with the conditions which will be fully defined by surface area and resistivity measurements.

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VII. GLOSSARY

1. Type A fiber - Fiber produced by a proprietary process ranging in mean diameter from 3 to 30 microns.

2. Type B fiber - Fiber derived from metal wool.

3. Type C fiber - Fiber derived from metal wire.

4. Fiber grade - Generally defines the fiber diameter. Since in type A and B the diameter is not constant, the grade designation is preferred to a mean diameter designation.

5. Fiber type and grade

   To avoid cumbersome discussions the type and grade of fiber used in a given specimen will be reduced to such terminology as AX1, meaning type A fiber Grade X1.

VIII. DISTRIBUTION LIST

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